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FINAL REPORT

An XPS Study of the Formation of Ultrathin

Platinum and Iridium Silicide Layers on
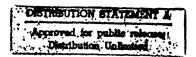
Si(100) and Si(111) Surfaces

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An XPS Study of the Formation of Thin Pt and Ir Silicide Overlayers on Si(100)2x1 Surfaces

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Abstract

The formation of thin (5-100Å) Platinum and Iridium silicide films on the clean Si(100)2x1 surface has been studied using XPS. A detailed analysis of the Si 2p and Pt/Ir 4f core-level photoemission lineshapes and intensities has allowed the determination of the phase formation sequence, and the resulting change in Schottky barrier height, with annealing. Progressively silicon rich silicides were formed with increasing temperature, at temperatures >600°C this process was limited by rapid surface segregation of silicon atoms from the substrate. Small changes in relative Schottky barrier height were observed for different silicide phases, with the monosilicide phases giving the lowest Schottky barriers.

1 Introduction

To understand quantitatively the electrical properties of transition metal (TM) silicide and TM / silicon interfaces it is necessary to gain insight into the chemical, electronic and structural properties of the interfaces. Silicide contacts formed using platinum and iridium are known to give the highest Schottky barrier heights to n-type silicon, this property makes such contacts useful in microelectronic and infra-red imaging technologies [1,2] and interesting also from a fundamental scientific viewpoint [3].

Core-level photoemission spectroscopy, using X-ray and synchrotron radiation, is an established technique for monitoring Schottky barrier formation and chemical reactions [4]. An X-ray photoelectron spectroscopy system, capable of resolving the Fermi level shifts (related to band bending) from chemical core-level shifts (related to chemical reaction) of the Si 2p core-level, has been used to study platinum and iridium silicide phase formation on clean Si(100)2x1 surfaces. This approach allows changes in Schottky barrier height and silicide phase to be measured concurrently using one analytic technique.

Previous work on the formation of platinum and iridium silicide thin films have shown that a range of silicide stoichiometries are possible [5-9]. In the first instance, platinum shows metal rich Pt_2Si formation at low temperature followed by PtSi formation at higher temperature (400°C) [5,6]. Iridium silicide follows a similar reaction path with IrSi forming at room temperature. For thin iridium films on silicon annealed at 300°C some dispute exists about whether $IrSi_{1.6}$ or $IrSi_{1.75}$ is produced [7,8] ($IrSi_{1.75}$ is reported to be a semiconducting compound [9]). At higher annealing temperatures, ~1000°C, $IrSi_3$ is reported to form [7]. The Schottky barrier heights of TM silicide / silicon systems change with changing silicide stoichiometry [3]. The aim of this work is to correlate changes in chemical phase of platinum and iridium silicides with changes in Schottky barrier height on clean Si(100)2x1 surfaces. (Note that $IrSi_{1.5}$, $IrSi_{1.6}$, and $IrSi_{1.75}$ are equivalent to Ir_2Si_3, Ir_5Si_8, and Ir_3Si_5 respectively).

Chemical core-level shifts have proved useful in establishing Schottky barrier models based on an interface bonding approach [10], the results of the data analysis are also discussed in this context.

The layout of the paper is as follows, Section 2 describes the experimental set-up used, the results are presented in Section 3 and discussed in relation to previous work, concluding remarks are made in Section 4.

2 Experimental

The experiments were conducted in an oil diffusion pumped Vacuum Generators MarkII Escalab. The UHV system was comprised of three chambers, an analysis chamber, a preparation chamber with a UHV load-lock, and an evaporation chamber. Samples, evaporation sources, and quartz

crystal film thickness monitors were mounted on portable stubs that could be introduced into the system via the rapid entry load-lock. All the chambers had a base pressure of $< 2 \times 10^{-10}$mb for the experiments. The surface science instruments utilised in the analysis chamber were a Mg Kα X-ray source, and a three channel hemispherical analyser with a resolution of 2% of the pass energy. The total instrumental resolution using a 5eV analyser pass energy was 0.7eV. The preparation chamber housed a rear-view LEED system for monitoring surface reconstructions, and a railway track for transferring the samples, evaporation sources, and film thickness monitors. The evaporation chamber contained a stage for mounting the evaporation sources, a manipulator for holding the sample holders and quartz crystal film thickness monitor.

The Si(100) samples were cleaned by annealing at 850°C for 20 mins, samples thus treated produced sharp two domain 2x1 LEED patterns and broad XPS scans showed no trace of oxygen or carbon contamination. The low annealing temperature ensured that problems with shallow surface doping by contaminants did not occur [11].

Platinum evaporation sources were constructed using a tungsten filament loaded with 99.9% pure platinum wire. After thorough outgassing slow evaporations were performed at pressures no higher than 1×10^{-9}mb. Iridium depositions were more difficult, 99.9% pure iridium wire (0.25mm dia.) filaments were used that gave low evaporation rates by sublimation when resistively heated. This method tended to have somewhat higher outgassing rates than platinum during evaporation, the maximum pressures being $\leq 1 \times 10^{-8}$mb for short periods.

Sample annealing temperatures were determined using two methods, by sample current measurement (previously calibrated in another vacuum system using a copper/constantin thermocouple) for low temperatures, and an optical pyrometer for high temperatures.

3   Results and Discussion

3.1   Data Analysis Procedures

The XPS core-level spectra were analysed using a least squares curve fitting program. The core-level photoemission peaks were fitted using Voigt functions (a Gaussian instrument response function convolved with the intrinsic Lorentzian core-level lineshape). The secondary electron background was subtracted using the Shirley function [12] for metal peaks and a cubic function the Si 2p peaks. The curve fitting routine was used to analyse up to three core-level doublets, which gave their intensities, positions, asymmetries etc. The lineshape analysis allows band bending and chemical reactions to be monitored at the same time.

The presence of surface states at the clean Si(100)2x1 surface pins the Fermi level and causes band bending near the surface. For the 3Ωcm

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material used in this work the band bending extends for about a micron into the substrate. The inelastic mean free path of the Si 2p photoelectrons excited using Mg Kα X-rays is about 25Å [13]. Therefore, as the photoelectron escape depth is much smaller than the depletion width, the binding energy of the Si 2p core-level peak can be used to monitor substrate band bending [4]. The Fermi level of the clean p-Si(100)2x1 surface is reported to be pinned at 0.35 eV above the valence band maximum [14]; contacts formed with the high work function metals platinum and iridium cause the Fermi level position to be pinned closer to the valence band maximum—thus reducing the band bending. Changes in band bending at the silicon surface caused by the presence of a platinum or iridium overlayer will cause a lowering of the Si 2p binding energy. Also, changing the chemical environment of the silicon atoms causes a change in the electronic potential surrounding such atoms, which in turn give rise to chemical core-level shifts. Si 2p XPS spectra measured for bulk silicides have shown that the silicide core-level chemical shifts occur at a higher binding energy than the bulk silicon [15], therefore, for the analysis of data utilising p-type silicon surfaces it is easy to differentiate between Fermi level and chemical shifts. (For n-type samples, of course, the Fermi level and chemical shifts are in the same direction—which makes the data analysis more difficult.)

The intensities of the core-level photoemission peaks can be used to determine the chemical state of a compound. The intensity of a core-level peak is dependent on the number of atoms being illuminated by the X-ray beam, the escape depth of the photoelectrons concerned, and the photoemission cross-section. Given that the photoemission signal comes solely from the compound, it is easily shown that the relative concentration of atoms present in a single phase of the compound is given by:

$$r = \frac{I_{2p}\lambda_{4f}\chi_{4f}}{I_{4f}\lambda_{2p}\chi_{2p}} \qquad eqn(1)$$

where I is the intensity of the core-level, λ is the electron escape depth of the core-level photoelectrons, and χ is the photoemission cross-section. Data tables for photoemission cross-sections were used in the data analysis [16], and photoelectron escape depths were estimated using the formalism devised by Seah and Dench [13]; these parameters are given in Table 3.1. Equation 1 can be used to determine the chemical state of thin silicide films from XPS data, provided stoichiometric compound formation has occurred and given sufficient resolution to resolve chemically shifted core level components. Given these conditions, the r value determined using the intensities of reacted core-level components of a silicide, can be used to determine the phase of a reacted species using:

$$MSi_r \qquad eqn(2)$$

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In this work, the intensity of the reacted Si 2p and metal 4f core-level peaks were used to determine the chemical phase of the silicides formed.

3.2 Platinum Silicide Formation

Two types of platinum / silicon experiments were performed, one with a thick 100Å platinum overlayer and the other concentrating on thin 5–20Å overlayers. XPS spectra were recorded for a range of annealing temperatures. The curve fitted data parameters are shown in Table 3.2.

After deposition of 100Å of platinum onto the clean Si(100)2x1 surface the photoemission features associated with the silicon substrate are attenuated (see Figures 1 and 2), so no information on Fermi level pinning can be obtained for this experiment. The Pt 4f core-level XPS spectra are representative of polycrystalline platinum, no silicon, oxygen or carbon features were observed indicating pure platinum evaporations. The pure platinum $4f_{\frac{7}{2}}$ emission is centred at 70.80 eV binding energy. After annealing at 350°C photoemission from the Si 2p core level is observed, and the predominant $Pt\ 4f_{\frac{7}{2}}$ emission shifts +1.70eV to higher binding energy. However, photoemission from from unreacted platinum is still observed at 70.80eV. Also, the emission from the Si 2p core-level is displaced from the value expected for elemental Si 2p by +1.05eV—clearly major atomic interdiffusion has occurred.

The half width half maximum (HWHM) of the $Pt\ 4f_{\frac{7}{2}}$ remains unchanged after annealing—indicating that the emission centred at 72.50eV is from single phase silicide. Comparing the relative intensities of the reacted Si 2p and Pt 4f core-levels using *eqn* 1 yields an r value of 1.0, indicating the formation of PtSi. At this stage of the annealing procedure the silicide layer is composed of PtSi together with a small quantity of unreacted platinum

Further annealing produces no further changes until the temperature reaches ~650°C when emission from the elemental Si 2p binding energy appears at 98.90eV, this becomes more pronounced at 750°C. Also, the $Pt\ 4f_{\frac{7}{2}}$ shows a rapid decrease in intensity. Clearly, outdiffusion of silicon atoms onto the surface has occurred that attenuates the signal from the silicide layer. Thus, in this experiment, any further silicide reactions are impossible to measure using XPS.

For the thin film experiments 5–20Å films were deposited onto clean Si(100)2x1 surfaces that were subsequently annealed to promote platinum / silicon reaction.

Typical Si 2p and Pt 4f core-level XPS spectra are shown in Figures 3 and 4. The clean Si 2p emission shows an slightly asymmetric peak with the maximum intensity centred at 98.90eV. (The asymmetry of the Si 2p photoemission peak caused by the spin orbit splitting of 0.61eV that is not fully resolved.)

After deposition of 5Å of platinum the Pt 4f spectrum shows a single

component centred at 70.90eV. The Si 2p spectrum shows large changes with a chemically shifted component centred at 99.40eV and a shift in the binding energy of the bulk Si 2p core-level. The Si 2p core-level photoemission from the silicon substrate shows a shift of 0.25 eV to higher binding energy, indicating a Fermi level shift of −0.25eV with respect to the clean Si(100)2x1 Fermi level pinning position. The relative intensity ratio of the Si 2p and Pt 4f components show that Pt_2Si formation occurs for Pt / Si(100) interface formed at room temperature.

Annealing this structure at 350°C promotes further chemical shifts. The Pt 4f emission is now centred at 72.50eV (the same as PtSi— determined in the thick film experiment) and the Si 2p spectrum exhibits a new component centred at 99.90eV. The relative intensities of the reacted core-level components of 1.0, and the magnitude of the binding energies of the Si 2p and Pt 4f core-levels indicate the presence of PtSi. A further decrease in the binding energy of the bulk Si 2p core-level component also occurs, this is consistent with further Schottky barrier lowering of the order of −0.05eV.

Further annealing at higher temperatures produces a large increase in the elemental Si 2p peak intensity and a rapid decrease in the Pt 4f peak intensity indicating that silicon outdiffusion has started at a lower temperature than for the thick film experiment.

3.3 Iridium Silicide Formation

Owing to the difficulties with iridium deposition only thin film studies were able to be conducted. The experimental procedure was identical to the thin film platinum / silicon experiments. The Si 2p and Ir 4f core-level spectra are shown in Figures 5 and 6, and the curve fitting parameters are given in Table 3.3. The pressure during iridium deposition rose to 1×10^{-10}mb which made oxygen and carbon contamination a problem.

After a 7Å iridium deposition onto the clean Si(100)2x1 surface major changes are observed in the Si 2p core-level lineshape. A Fermi level shift of the bulk Si 2p component from 98.90eV to 98.70eV to lower binding energy is observed, and a chemical core level shift at 99.20eV binding energy is seen. The Ir $4f_{\frac{7}{2}}$ peak is situated at 60.50eV binding energy, the same as the tabulated value of the Ir $4f_{\frac{7}{2}}$ binding energy [17] (after correction for the difference in analyser work function). However, in this analysis there is no bulk Ir $4f_{\frac{7}{2}}$ to compare with to determine whether there is any broadening of the peak. However, the HWHM of 0.60eV is the same to that of the pure Pt $4f_{\frac{7}{2}}$ peak and remains unchanged after annealing—implying that the iridium atoms are situated in a single phase system. The intensity ratio of the reacted Si 2p component and the Ir $4f_{\frac{7}{2}}$ peak indicate IrSi formation in agreement with previous studies [7], reinforcing the assertion that the chemical environment of the iridium atoms is a single phase.

Annealing at temperatures between 300°C and 500°C produced an Ir $4f_{\frac{7}{2}}$ shift of +0.30eV to 60.80eV binding energy. A Si 2p reacted component is seen at 99.30eV in conjunction with a Fermi level shift of the bulk Si 2p of +0.05eV. The intensity ratio of the Si 2p chemical shift and the Ir $4f_{\frac{7}{2}}$ peak gives an r value of 1.6 that has an estimated uncertainty in r of ±20%. This observation is in agreement with reference [7], however, given the uncertainty in r (and the contamination problem) the presence of $IrSi_{1.75}$ (or a mixture of the two phases) cannot be commented upon. The +0.05eV Fermi level shift is consistent with an increase in the formation of the Schottky barrier height after $IrSi_{1.6}$ formation.

Previous studies [7] have reported that $IrSi_3$ forms at annealing temperatures of ~1000°C. However, in this study no further chemical shifts of the core-levels were observed after annealing 5–20Å iridium films on the Si(100)2x1 surface at temperatures between 600°C and 1000°C. The only change that was observed was a rapid increase in the Si 2p signal (with a corresponding decrease in the Ir 4f intensity) caused by silicon segregation to the surface—which attenuates the photoemission features associated with reactions in the silicide layer.

3.4 Discussion

The phase formation sequences of thin film platinum and iridium silicide on Si(100) substrates exhibit similar trends, with both systems show increasing silicon content with annealing.

The results for platinum silicide formation are largely in agreement with other studies [5,6,15], however, a UPS / Auger study by Matz et al. inferred [18] the existence of an intermixed silicide-like phase for the formation of the platinum / silicon interface at room temperature, as opposed to the single phase Pt_2Si in this work. The disparity between studies of low temperature platinum / interface interface formation has been noted by Rossi [15] who attributed any variations in results as being caused by the uncontrolled kinetics of the interface formation.

The observation that thin platinum films deposited on Si(100)2x1 surfaces form Pt_2Si implies that thick platinum films (~100Å) form a bilayer structure, comprising of a reacted silicide interfacial layer and an unreacted platinum overlayer. The ultimate stoichiometry of a reacted interfacial layer cannot be commented upon as XPS spectra cannot provide information on interfaces which are buried at a depth much greater than the photoelectron mean free paths. Unfortunately, because of the absence of any signal from the silicon substrate nothing can be inferred about the Fermi level pinning position at the interface.

The phase formation sequence of iridium silicides has also been the subject of some dispute [7,8]. The silicide formed at room temperature is generally agreed to be the monosilicide IrSi—for thin films, as found in this study. Thicker iridium films (~100Å) give a bilayer structure

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composed of an IrSi interfacial layer and an unreacted iridium overlayer [7], which is somewhat similar to the behaviour of the room temperature platinum / silicon interface.

Weiss et al. [8] reported the formation of Ir_2Si and $IrSi_3$ for coevaporated iridium–silicon films on SiO_2 substrates, the SiO_2 does not supply silicon for the silicide reaction allowing the stoichiometry of the silicide to be controlled by the relative concentrations of silicon and iridium atoms in the coevaporated layer. However, for the clean Ir / Si(100) experiments performed in this work there is an unlimited supply of silicon atoms available for reaction from the substrate, which prevents metal–rich silicide formation. The formation of thin film $IrSi_3$ was not observed because of rapid segregation of silicon atoms onto the surface at temperatures greater than $\sim$650°C, this problem was not mentioned by Wittmer et al. [7] who reported $IrSi_3$ formation at $\sim$1000°C. It may be possible to circumvent this problem using rapid thermal annealing to form $IrSi_3$ before a significant quantity of silicon has diffused to the surface.

The Schottky barrier trends of platinum and iridium silicide are also similar with the monosilicide phases producing the largest band bending change. Relating the observed band bending changes to absolute Schottky barrier heights is problematic because of the uncertainty in the initial Fermi level pinning position. However, the technique is accurate enough to compare relative Schottky barrier heights. These results show that the lowest barriers to p-type (equivalent to the highest barriers on n-type material) Si(100)2x1 were produced by the monosilicide PtSi and IrSi phases.

4 Conclusions

XPS has been used to monitor the phase formation sequence and Schottky barrier formation of platinum and iridium silicides on Si(100)2x1 surfaces. The platinum phase formation sequence was found to be Pt_2Si at room temperature with PtSi formation occurring above 350°C. For iridium overlayers, IrSi was found to form at room temperature. Annealing at 300°C–600°C produced an $IrSi_{1.6}$ compound—although the presence of an $IrSi_{1.75}$ compound could not be entirely ruled out. Attempts to produce further silicon-rich silicides by high temperature annealing were unsuccessful because of problems with surface segregation of silicon atoms. Band bending shifts of the substrate Si 2p core-level, during silicide formation, show that the lowest barriers on p-type silicon (highest on n-type material) were produced by the monosilicide phases.

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Table 1   Data Analysis Inelastic MFP's and Photoemission

Cross-sectons.

Core-level	Kinetic Energy (eV)	Inelastic mfp[1] (Å)	Cross-section[2] (Mb)
Si 2p	1154.7	24.5	0.02
Pt 4f	1182.8	24.8	0.40
Ir 4f	1193.1	24.9	0.35

1. Calculated using the formalism in reference [13].

2. From reference [16]

Table 2   Curve-fitting Results for Platinum / Silicon

Reaction Temperature	Core-Level	Chemical Shift (eV)	Fermi Shift (eV)	Silicide phase
Room Temperature	Si 2p	+0.50	-0.25	Pt_2Si
	Pt 4f	+1.00	—	
350°C	Si 2p	+1.00	-0.30	PtSi
	Pt 4f	+1.70	—	
500°C	Si 2p	+1.00	—	PtSi + segr-egation
	Pt 4f	+1.70	—	
>600°C	Si 2p	segreg-tion		segre-gation
	Pt 4f		—	

Pt $4f_{\frac{7}{2}}$ Binding Energy = 70.80eV

pSi(100)2x1 $2p_{\frac{3}{2}}$ binding Energy = 98.90eV

Table 3   Curve-fitting Results Iridium / Silicon

Reaction Temperature	Core-Level	Chemical Shift (eV)	Fermi Shift (eV)	Silicide phase
Room Temperature	Si 2p	+0.30	-0.20	IrSi
	Ir 4f	0.00	——	
350°C	Si 2p	+0.40	-0.15	IrSi$_{1.6}$
	Ir 4f	+0.30	——	
500°C	Si 2p	+0.40	-0.15	IrSi$_{1.6}$
	Ir 4f	+0.30	——	
>600°C	Ir 2p	segrega-tion	——	segreg-ation
	Pt 4f		——	

p-Si(100)2x1 2p$_{\frac{3}{2}}$ Binding Energy = 98.90eV

Ir 4f$_{\frac{7}{2}}$ Binding Energy = 60.50eV

Pt/Si(100)2x1

750C Anneal

650C Anneal

500C Anneal

350C Anneal

Intensity (Arb. Units)

Binding Energy (eV)

Pt/Si(100)2x1

Intensity (Arb. Units)

750C Anneal

650C Anneal

500C Anneal

350C Anneal

Room Temperature

Binding Energy (eV)

65 67 69 71 73 75 77 79 81 83 85

Pt/Si(100)2x1

750C Anneal

550C Anneal

350C Anneal

RT Deposition

Clean Si(100)2x1

Intensity (Arb. Units)

Binding Energy (eV)

Pt/Si(100)2x1

Intensity (Arb. Units) vs **Binding Energy (eV)**

750C Anneal

550C Anneal

350C Anneal

Room Temperature

Ir/Si(100)2x1

850C Anneal

550C Anneal

RT Deposition

Clean Si(100)2x1

Intensity (Arb. Units)

Binding Energy (eV)

Ir/Si(100)2x1

850C Anneal

550C Anneal

Room Temperature

Intensity (Arb. Units)

Binding Energy (eV)